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Measurements of Shock Compressibility of C_{70} Fullerene with the Use of Time-Resolved Synchrotron Radiation Technique

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Abstract: First-time determination of shock compressibility of C_{70} fullerene was performed with the use of time-resolved synchrotron radiation technique. Starting C_{70} specimens were prepared by high (1 GPa) hydrostatic pressure treatment of polycrystalline C_{70} with a hexagonal closed-packed structure. The specimens consisted of the hexagonal closed-packed (70s vol.%) and the rhombohedral (30 vol.%) crystalline phases due to an incomplete phase transition of the hexagonal phase to the rhombohedral phase which occurred in the course of preparation of the specimens. The density of the specimens was 1.65 g cm^{-3}, the X-ray density of the specimens was 1.663 g cm^{-3}. To provide the experiments, VEP-1 particle accelerator of Budker Institute of Nuclear Physics was used as a pulsed-periodical source of synchrotron radiation. Hugoniot of C_{70} was determined at pressures of 6.3-9.3 GPa and is situated at a position lower than the Hugoniot of C_{60} fullerene on pressure-specific volume plane. Any peculiarities of shock compression curve that could be connected to polymerization of fullerene C_{70} or to formation of a hard carbon phase were not detected in the explored pressure range (in contrast to the shock behavior of fullerene C_{60} in the same pressure range). The data on shock compressibility of C_{70} fullerene can be used for construction of its equation of state and for smart forecasting of shock response of affordable C_{60}/C_{70} mixes and of metal-fullerene composites.

Key words: Fullerene C_{70}, shock compressibility, synchrotron radiation

INTRODUCTION

By this time thermodynamic properties and phase transformations of C_{70} fullerene at high pressures are not well understood. Main part of the available experimental data were collected at high static pressures with the use of diamond anvil cells and high-pressure chambers of different types and were reviewed by Sundqvist (1999). On the basis of these data, the equilibrium phase diagram of C_{70} was offered (Sundqvist, 1999). With relation to this diagram, C_{70} is considered as an individual molecular compound, because C_{70} is one of the least stable carbon allotropes (the enthalpy of formation of C_{70} is 3069 kJ kg^{-1} (Diogo *et al*., 1997). All C_{70} phases marked on this diagram (the low-temperature monoclinic, the rhombohedral and the high-temperature face centered cubic) are molecular crystals. The C_{70} phase with a hexagonal closed-packed structure is known, too. According to Sundqvist (1999) this phase is metastable overall the phase diagram.

Previously, phase transformations of C_{70} under stepwise shock compression were experimentally studied with the use of recovery assemblies (Milyavskiy *et al*., 2005a, 2008). Shock-induced transformation of the hexagonal phase of C_{70} into the face centered cubic phase of C_{70} was observed at pressures in the range 9-23.5 GPa. The amount of transformed material increases with the shock intensity. Upon further increase of the shock pressure the destruction of C_{70} molecules occurs. This destruction is accompanied by a formation of graphite-like carbon.

In this work shock compressibility of C_{70} fullerene was measured. The measurements were performed with the use of VEP-1 particle accelerator of Budker Institute of Nuclear Physics of Siberian Branch of Russian Academy of Sciences as a pulsed-periodical source of Synchrotron Radiation (SR). Preliminary results of these measurements were presented by Milyavskiy *et al*. (2009). It is the first example of using this experimental technique for studies of shock waves in inert solids. The applicability of SR for studies of detonation processes and shock waves in inert high porous media (silicon dioxide aerogels) was demonstrated previously (Ten *et al*., 2005; Merzhievsky *et al*., 2009).

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MATERIALS AND METHODS

In the experiments, polycrystalline C_{70} powder with purity not less than 99.5% was used. The powder has been received from Joint stock company Fullerene centre (http://www.fullerene-c.com). The X-ray powder diffraction study of the starting powder showed that the material consists of C_{70} molecular crystals with a hexagonal closed-packed structure. The starting C_{70} powder was pressed with the use of a special piston-cylinder type apparatus (made of beryllium bronze alloy) and a hydrostatic pressure chamber by the pressure of 0.22 GPa. Then the tablets produced by this way were subjected to high (1 GPa) hydrostatic pressure treatment with the use of the same hydrostatic pressure chamber. Finally, the starting C_{70} specimens had a density of 1.65 g cm^{-3}, a longitudinal sound velocity of 2.1 km sec^{-1}, a diameter of 15 mm and a thickness of 2.5-3.5 mm. To clarify the phase composition of the starting specimens, the microstructure of the specimens was studied with the use of X-ray powder diffractometry. The specimens consisted of two crystalline phases. The first phase (70s vol.%) had a hexagonal closed-packed structure (lattice parameters a = 1.062 nm, c = 1.734 nm, number of C_{70} molecules per elementary cell z = 2) and the second phase (30 vol.%) had a rhombohedral structure (lattice parameters refereed to hexagonal axes a = 1.010 nm, c = 2.793 nm, z = 3). Thus, an incomplete phase transition of the hexagonal phase to the rhombohedral phase occurred as a result of action of high static pressure in the course of preparation of the specimens. The ideal density of these specimens based on the lattice parameters and the phase composition was ρ_0 = 1.663 g cm^{-3}, thus the specimens were slightly porous.

Specimens were loaded by plane impacts of aluminum 2024 flyers accelerated by products of detonation of the high explosives through the air gap. The diameter of flyers was 16 mm. Explosive projectile systems (Merzhievsky *et al*., 2009) were used for driving flyers (Fig. 1). The system consisted of a plane shock wave generator and a tablet (weight of a tablet was 20 g) of high explosives (a mixture of trinitrotoluol and cyclonite 50/50). The velocity acquired by the flyer depending on its thickness was 1.3-1.8 km sec^{-1}.

The experimental procedure similar that which was described by Merzhievsky *et al.* (2009) was used to determine parameters of shock waves generated in the specimens by impact of the flyers. This procedure is based on the visualization of the movement of density discontinuities. Positions of discontinuities are determined by measuring the degree of the attenuation of SR in an examined specimen during passage of compression and rarefaction waves through that specimen. The positions were evaluated each 250 nsec. For this purpose each 250 nsec cultrate SR beam came into the explosive chamber through narrow slit covered by beryllium window, passed through the experimental assembly, then passed through another beryllium window and came to SR detector. SR beam has a width of 18 mm, a thickness of 0.4 mm and duration of 1 ns. The linear (one-dimensional) DIMEX SR detector (Aulchenko *et al*., 2003) was used. The detector was placed parallel the axis of the assembly at a distance of 770 mm from it. That linear detector consisted of 256 sensitive elements. Each element has a height of 1 mm and a length of 0.1 mm. The total length of the linear detector along the axis is 25.6 mm. The initiation of the detector occurred just when the contact gauge (mounted between the detonator and the plane shock wave generator) was shorted out by the products of detonation.

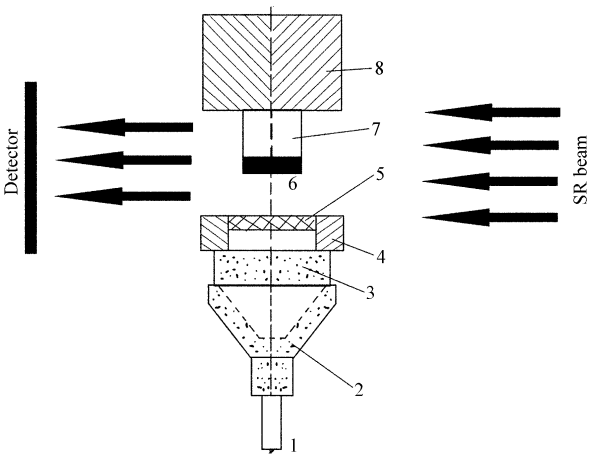

Fig. 1: Scheme of the experimental assembly: 1: Detonator; 2: Plane shock wave generator; 3: High explosives; 4: Focusing ring; 5: Aluminum 2024 flyer; 6: Specimen; 7: Base plate (polymethylmethacrylate or aluminum); 8: Support

RESULTS AND DISCUSSION

The typical experimental distance-time diagram is presented in Fig. 2. This diagram allows us to calculate the velocity of flyer, the velocity of shock wave front in the specimen, the velocity of flyer-specimen interface. To calculate thermodynamic quantities (for example, pressure and specific volume), standard relations conserving mass and momentum across the shock front (Kanel *et al*., 2004) and etalon Hugoniot of aluminum 2024 ($P = 2.784 \times (5.349 + 1.317 \times U) \times U$, where P is the pressure behind the shock wave front (in GPa), U is the mass velocity behind the shock wave front (In km sec^{-1}) were

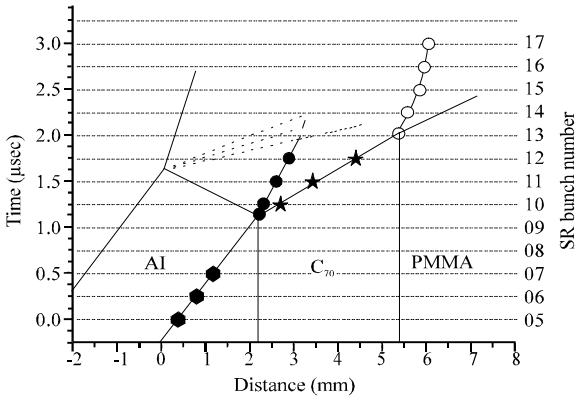

Fig. 2: Experimental distance-time diagram. The flyer moves left to right. The base plate is made of polymethylmethacrylate (PMMA). Markers indicate positions of density discontinuities at the consecutive moments of time: Solid circles-the positions of flyer-specimen interface; solid stars the positions of shock wave front in the specimen; open circles-the positions of the specimen-base plate interface. Dotted lines represent the wave of adiabatic unloading

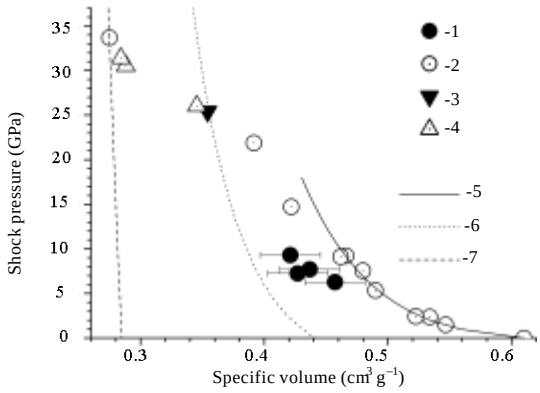

Fig. 3: Hugoniot of C_{70} fullerene on pressure-specific volume plane (1). Experimental Hugoniot of C_{60} fullerene (2-4; 3 and 4-parameters of first and second shocks of two-wave structures, respectively). Hugoniots (ρ_{00}=1.64 g cm^{-3}) calculated with the use of EOS of *sc*-phase of C_{60} fullerene (5), graphite (6) and diamond (7)

used. This Hugoniot was constructed by approximation of aluminum 2024 experimental data (Marsh, 1980) in the range of 0.278 km sec^{-1} < U < 2.095 km sec^{-1}.

Results of measurements of shock compressibility of C_{70} fullerene are presented in Fig. 3. In Fig. 3, experimental Hugoniot of C_{60} fullerene (Milyavskiy *et al*., 2005b) is shown, too. Also, theoretical Hugoniots of C_{60} fullerene (the simple cubic phase), porous graphite (with a starting density of 1.64 g cm^{-3}) and porous diamond (with a starting density of 1.64 g cc^{-1}) are presented. These Hugoniots were computed with the use of Equations of States (EOS) (Khishchenko *et al*., 2005, 2007). It is clear that the experimental Hugoniot of C_{70} fullerene in the studied range of pressure (6.3-9.3 GPa) is situated at a position lower than the experimental Hugoniot of C_{60} fullerene (Milyavskiy *et al*., 2005a) on the pressure-specific volume plane. Any peculiarities of shock compression curve that could be connected to polymerization of fullerene or to formation of a hard carbon phase were not detected in the explored pressure range (in contrast to the shock behavior of fullerene C_{60} in the same pressure range (Milyavskiy *et al*., 2005b).

CONCLUSION

The shock response of C_{70} fullerene was studied at pressures in the range 6.3-9.3 GPa and compared to the shock response of C_{60} fullerene in the same pressure range. The data on shock compressibility of C_{70} fullerene in future will be used for construction of its equation of state just as it earlier has been made for fullerene C_{60} (Khishchenko *et al*., 2007). Also, these result together with prior experimental data on phase transformations of C_{70} fullerene under stepwise shock compression and with the results on shock compressibility and shock-induced phase transitions of C_{60} fullerene (Milyavskiy *et al*., 2005b) will be used for smart (i.e., characterized by real understanding of a role of each of components) forecasting of shock response of commercially available mixes of fullerenes C_{60} and C_{70} (these mixes are much more affordable then pure fullerenes) and of metal-fullerene composites which are developing on the basis of these mixes (Milyavskiy, 2008).

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